

# Alaska Scientific Crime Detection Laboratory

## Controlled Substances Procedure Manual

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## SECTION 1 INTRODUCTION

### 1.1 OVERVIEW

The Controlled Substances discipline performs analysis of drugs, including controlled substances, pharmaceuticals, and clandestine lab samples. The majority of case submissions involve the identification of controlled substances. In these cases the *objective* of the Forensic Scientist is to conclusively identify controlled substances in a sample or conduct sufficient analysis to determine that no controlled substances are detected.

Controlled substances are those substances designated by the legislature of the State of Alaska in the Alaska Statutes 11.71.

'Controlled Substance' means a drug, substance, or immediate precursor included in the schedules set out in AS 11.71.140 - 11.71.190.

Additionally, Forensic Scientists may be called upon to analyze samples for federal agencies operating within Alaska for substances controlled under the Uniform Controlled Substances Act. However, this laboratory does not perform purity (quantitative) testing and does not routinely sample multiple items based on federal threshold levels.

### 1.2 SAMPLE PRIORITIZATION

In order to provide timely service and meet the mandates of the Laboratory's Performance Metrics as reported to the Department of Public Safety, the Scientific Crime Detection Laboratory has adopted policies in the Controlled Substances discipline involving the prioritization and selection of evidence analyzed:

- a. If weighable quantities of material are present, residues may not be analyzed. Exceptions are multiple suspects, concurrent fingerprints, or the item being the reason for a search warrant, but this information must be communicated to the laboratory.
- b. If multiple quantities of materials are submitted, one exemplar from each type of item may be analyzed (for example: if 3 bags plant material, 3 bags of chunky material, and 3 bags of powder are submitted, then 1 bag each of plant material, chunky material, and powder may be analyzed)
- c. If there are multiple suspects, the submitter must indicate which items are associated with which suspects to ensure analysis.
- d. For each submission of evidence on a case (each Request for Lab Services), there will generally be ONE report for each type of testing. If there are multiple offense dates or multiple suspects, and separate reports are needed, the evidence should be submitted on separate lab requests. (This is most common with federal investigations.)

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### 1.3 SAMPLE CONSERVATION

A portion of the original sample will be left in order to allow for subsequent retesting or for defense purposes. In cases where only residue amounts are submitted, that residue must be a visible amount, sufficient for reanalysis.

In the event of invisible or residue amounts of evidence, the analyst should not analyze. These instances would be reported as "Quantity Insufficient for Analysis" with details recorded on the analyst's worksheet.

When sufficient analytical data exists to suggest that a substance is present, but insufficient sample exists to definitively identify the substance, "Insufficient Concentration for Identification" will be reported.

Sample vials may be retained in cases where other laboratory analyses may prevent future testing of remaining trace evidence. While retaining the sample vial is an attempt to preserve the sample, it is not a guarantee against sample degradation or leakage.

### 1.4 IDENTIFICATION REQUIREMENTS

A conclusive identification of a drug substance (whether controlled or uncontrolled) will include analysis by infrared or mass spectroscopy, instrumentation that characterizes the molecular structure of a compound. GC/MS, though a composite of two determinations, is considered as one test since it involves one sampling of the evidence. A second sampling or independent identification of a drug substance is required. This second sampling, usually done as presumptive testing, can be any of the protocols discussed in this manual:

- a. Literature reference
- b. Color test
- c. Microcrystalline test
- d. GC/MS (if a second test, must be a second sampling)
- e. FTIR

See Section 2.7 for guidelines on the analysis of general drug substances.

NOTE: Identification of non-drug elemental substances such as Iodine and Phosphorus is exempt from the requirement for two samplings and a spectrally elucidating procedure.

NOTE: Identification of a non-controlled drug (e.g. acetaminophen, ibuprofen, guaifenesin, or diphenhydramine) in a pharmaceutical preparation is exempt from the requirement that a standard be analyzed concurrently. When identification may be important in determining the charging statute, a library search of previous GC/MS spectra along with a literature search of the pharmaceutical is sufficient.

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Marijuana (Cannabis) is identified through physical plant characteristics (botanical identification) and GC/MS (chemical identification). The GC/MS must demonstrate the presence of tetrahydrocannabinol. See Section 2.6 for more guidelines on the analysis of marijuana and marijuana products.

If used, color tests or microcrystal tests must provide a positive result for each controlled substance in a sample.

It is permissible to use the same instrumental technique on two separate samplings to identify a sample.

Reporting "No Controlled Substances per Alaska Statutes" may be accomplished by means of a single structurally-elucidating analysis (utilizing an appropriate screening program) OR by reference literature findings which allow identification of the drug as one that is uncontrolled in Alaska.

### Special Considerations:

- a. For salt vs. base determinations of cocaine, a generally accepted practice is to use infrared analysis.
- b. For samples indicating the presence of Clorazepate or Nordiazepam refer to the Clorazepate extraction procedure. These drugs cannot be identified by GC/MS and must be extracted and analyzed by FTIR.
- c. For mushroom cases a Weber test must be performed to distinguish between psilocyn and psilocybin.

The major limitation in the analysis of an item is the size and condition of the sample submitted for analysis.

### Literature References

1. Butler, W.P. "Methods of Analysis for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs", Internal Revenue Service Publication No. 341: Washington, D.C., 1967, pp.92-93.
2. Moffat, Osselton, & Widdop, "Clarke's Isolation and Determination of Drugs and Poisons", 3<sup>rd</sup> Edition, Pharmaceutical Press: London, 2004.

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## SECTION 2

## TECHNICAL PROCEDURES

### 2.1 VISUAL SCREENING

When numerous individual 'units' are included in an item of submitted evidence, an analyst is called upon to use his or her training, experience, and competence in determining the homogeneity of the samples. Once the number of populations is determined, a sampling plan can be employed to do presumptive and confirmatory testing.

- If the item is plant material, the analyst must be aware of state weight thresholds. Any units within the item that do not conform to the analyst's homogeneity expectations must receive a separate analysis according to protocols.
- If the item is not plant material (powder, tablets, blotter squares, etc.), the analyst must determine visually (to the best of his ability) how many populations are within the item, segregate the units within the item, and employ sampling protocols in the selection of items for analysis. See Section 2.9 for sampling protocols. If an item contains 25 baggies of white crystalline substance and 3 baggies of off-white powder, the analyst would first segregate the items into two groups (A and B) before sampling for analysis. All testing is recorded in the analyst's bench notes.
- Visual screening of multiple units to determine the number of populations present does *not* count as one of the two required tests for identification.

### 2.2 LITERATURE REFERENCE

Identification by a literature search refers to:

- The identification of pharmaceuticals in dosage unit form utilizing shape, color, and manufacturer's markings/imprints. Literature searches of pharmaceuticals are appropriate when:
  - a. a presumptive physical identification of a product or ingredients is sufficient before reporting a non-controlled substance, or
  - b. triaging of multiple items in a case has identified the pharmaceuticals as not apparently vital to the total prosecution, or
  - c. physical identification is done as a secondary test to chemical analysis when an absolute identification is required.

Literature searches on pharmaceuticals (tablets, pills, capsules) are *inappropriate* when:

- a. it is the only test and a conclusive identification is required, or
  - b. counterfeiting or mislabeling is suspected.
- Literature search also can refer to the identification of drugs that have been found in instrumental data by use of a reputable spectral search library, but no standard is available in the laboratory for confirmation (GC/MS) or standard protocol utilizes a search function library (FTIR).



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- The third use of the term 'literature reference' is a comparison of unknown spectra to published spectra from journals (MicroGram, CLIC), forensic drug laboratories (DEA, FBI), conference or symposium notes, or verified internet references (Erowid.org).

### **BENCH NOTES:**

- Pharmaceuticals
  - a. Analyst's notes should describe evidence pharmaceuticals in terms of dosage form, color, and markings.
  - b. Analyst's notes should describe the matching product in literature in terms of reference used, year of reference, active ingredients, and dosage.
  - c. Analyst's notes, when including a literature search, must
    1. Document the source of the information.
    2. If a scanned copy of an internet reference is included, label with BOTH the lab case number (unique identifier) and the analyst's initials.
- Spectra data (GC/MS or FTIR)
  - a. Analyst's notes when including a literature search, should include the name of the spectral library searched and a copy of the comparison between the evidence spectra and library spectra.
  - b. If the library is not electronically available for actual comparison to the data on the instrument, a copy of the citation (be it a MicroGram or CLIC journal, spectra from another crime laboratory's standard, or other approved source) should be labeled with the case number and analyst's initials and scanned into the case file.
  - c. As with any instrumental analysis, the worksheet should indicate that GC/MS or FTIR was performed, what extraction was done, and what results were found.

### **REPORT:**

- a. Pharmaceuticals: Non-controlled tablets identified by literature identification alone should be reported as "Xxxxx - physical identification only" (count and dosage form are not needed). If literature confirmation is in *addition* to a confirmatory chemical test, the report shall simply indicate the drug and amount.
- d. Other drugs: If the drug identified is not a controlled substance, the report will indicate "No Controlled Substances per Alaska Statutes Detected". If the presence of a non-controlled substance changes the schedule of another drug in the item which *is* a controlled substance, it is reported as is any other drug.

### **REFERENCES:**

The following are accepted as references for use in establishing physical identification of pharmaceuticals:

- a. Physician's Desk Reference (PDR)<sup>™</sup>, Medical Economics
- b. Logo Index<sup>™</sup>, DEA
- c. Identidex<sup>™</sup>, Micromedex Inc.



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- d. Drug Identification Bible™, Amera-Chem, Inc.
- e. Government and manufacturer's websites (A printed image of the pharmaceutical is required when using a manufacturer as a reference.)
- f. Ident-A-Drug™, Therapeutic Research Center
- g. Pharmer.org, Drugs.com, RxList.com, or other information websites
- h. Direct information from a pharmacist or manufacturer (Notes must indicate the source of the information.)

The following are accepted as searchable electronic library references for use in establishing the identification of drugs found by GC/MS when the laboratory lacks a chemical standard:

- a. Agilent "Identification of Synthetic Cannabinoids by GC/MS"
- b. SWGDRUG
- c. DEA
- d. NIST
- e. Wiley
- f. Alaska Scientific Crime Detection Laboratory in-house library (DRUG)

The following are accepted as non-electronic library references for use in establishing the identification of drugs found by GC/MS when the laboratory lacks a chemical standard:

- a. CLIC
- b. DEA MicroGram
- c. FBI Laboratory
- d. DEA Research and Testing Laboratory
- e. Other citations as approved

The following are accepted as references for use in establishing the identification of drugs found by FTIR:

- a. HR Georgia State Forensic Drugs
- b. Alaska Scientific Crime Detection Laboratory library

If there is any question as to the validity or appropriateness of the comparing citation, the Controlled Substances Supervisor, QA Manager, or Lab Manager should be consulted.

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### 2.3 QUANTITY DETERMINATION

#### WEIGHT

Mass weights are determined in metric units on top-loading balances. Actual balance readings are recorded in the analyst's notes and not rounded or truncated in those notes.

A net weight will be obtained on all powders, plant material, and physical substances when practical. Liquids may be weighed or a volume measurement obtained. Large seizures of liquid evidence may need to be estimated. Seizures of tablets or capsules will receive a count or weight at the analyst's discretion. If the weight is in such a form as to make weight determination difficult and time-consuming, such as a thin film of residue in a pipe (*"residue"* or *"N/A"*) net weights are not required.

The method of measurement, either net or gross, shall be indicated in the bench notes. All weights in the report are net weights unless otherwise indicated.

Proper weighing techniques:

- a. Place material into a tared container and obtain a net weight. This will accommodate most drug samples.  
Other options:
- b. Weigh material directly.
- c. Weigh the original container with its contents, empty the contents, weigh the empty container, and subtract the difference in the two weights (weight by difference). Analyst must show the subtraction in their notes.
- d. Obtain the net weights of individual items in an item and sum the individual weights (weight by summation). Analyst must show the individual weights and summation in their notes.
- e. For liquids, volume *or* weight may be reported. When weights or volumes are estimated, that information is communicated in the report.

For reporting purposes, weights will routinely be truncated to one decimal point (tenth of a gram). Weight measurements under 0.10 gram will be reported as less than a tenth of a gram or as " $<0.1$  g".

#### COUNT

Large quantities of pharmaceuticals may be weighed and the approximate number of units estimated.

NOTE: If a count of pharmaceuticals, blotter squares, or individual packages differs from what the submitting agency has recorded on the lab submittal, it is recommended that a second laboratory person perform a count; this person need not be competency tested in the Chemistry discipline. The verification of the count should be recorded on the worksheet.

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### 2.4 COLOR TESTS

The following color tests are conducted as described below. For preparation of reagents, see Section 6 REAGENT PROCEDURES. The actual amount of sample needed for a color test is dependent on the concentration of the drug and the sensitivity of the test to the drug in question. The analyst should use a minimal amount of material, especially if the sample size is small.

#### LIMITATIONS:

*False negatives* can occur if:

- The amount of drug is below the sensitivity of the test.
- A positive reaction is hidden by the presence of another substance that reacts more quickly or more intensely.

*False positives* can occur if:

- Contamination is present.
- The analyst misinterprets the color reaction due to other substances with a similar reaction.

Reagents are tested at the time of preparation to ensure that they are functioning properly, and the results are recorded in the Reagent Log Book. All reagents will be routinely re-tested every 6 months and the results recorded in the Reagent Log Book. Analysts may, at their discretion, analyze positive and negative controls concurrently with evidence undergoing testing. Since these are preliminary screening tests, this is not necessary.

The discipline supervisor will maintain a calendar as a reminder of reagent verification (January and July) and other scheduled audits (see Section 5.2 Standards, Security).

#### • MARQUIS TEST

Place 1-3 drops of Marquis Reagent in a clean white spot plate or test tube and add several particles (or dried liquid residue) of the sample; observe and record response. Opiates give a characteristic violet color, while amphetamines give a red/orange. Do not confuse legitimate color reactions with charring reactions from the sulfuric acid. Be careful to distinguish the red/orange color of amphetamines from the gold or dull brown color that sometimes occurs with ephedrine. Note that reactions are faster and more intense with fresh reagent.

violet	opiates (heroin, morphine, codeine, etc.)
gray to violet-black	MDA, MDMA, propoxyphene
yellow	diphenhydramine
orange to brown	amphetamine, methamphetamine, phentermine, mescaline
slow pink to rose	aspirin
yellow	methyldone (3,4-methylenedioxymethcathinone)

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- **SCOTT'S TEST (Cobalt Thiocyanate)**

Place sample in a small tube. Add approximately 5 drops of Scott's Reagent to sample and shake. Observe for the formation of blue color and/or precipitate. Cocaine salts give a clumpy blue precipitate while cocaine base gives no reaction at this step. Follow with the addition of approximately one drop of concentrated HCl and observe blue color disappear and the color turn to pink. Add several drops of dichloromethane and shake. The dichloromethane (lower) layer will develop an intense blue color if cocaine (salt or base) is present. Cocaine salt gives a positive test, which is blue/colorless or pink/blue dichloromethane.

**Comments:**

The Scott's test is commercially manufactured into a standard field test for law enforcement officers. This three step test distinguishes cocaine from such common adulterants as lidocaine, procaine, benzocaine, and tetracaine. Cocaine base does not give a positive reaction for this test. A modification of the Scott's Reagent using 10% acetic acid instead of water will successfully detect cocaine in either base or salt form.

- **ACIDIFIED SCOTT'S TEST (Cobalt Thiocyanate)**

This modification of the Scott's Test uses 10% acetic acid instead of water in the preparation of the cobalt thiocyanate solution. Since the acid converts base cocaine to salt, both forms will give a positive reaction to the first step of this test.

- **WEBER TEST (For Psilocyn in mushrooms)**

Add a small mushroom sample (or alcohol extract) to a clean spot plate. Analyze a psilocyn standard or a known psilocyn-containing mushroom fragment in a separate well. Use a blank well or negative mushroom sample for a negative control. Add Weber Reagent and look for a purplish-red color. Then add one drop of concentrated HCl. A navy blue color indicates psilocyn.

- **PARA-DIMETHYLAMINO BENZALDEHYDE TEST (p-DAB or Van Urk's)**

Although this reagent has its primary use as a thin layer chromatography color developing chemical for hallucinogens, it can also be used as a spot test for procaine and is reported to react with some synthetic cannabinoids in a procedure involving a methanol extract and heat.

Yellow  
Violet

procaine  
LSD, psilocyn, psilocybin

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- **STARCH TEST (for Iodine)**

Iodides may be identified by means of a well-known color reaction in which free  $I_2$  gives a blue color with starch. This may be accomplished by utilizing:

- a. a test tube of suspected iodine with a cotton plug which has been soaked in starch solution, or
- b. a white spot plate with suspected iodine in a well, covered with a microscope slide smudged with starch.

Gentle heating may speed the reaction. If item is an iodine tincture (iodide), addition of an oxidizing agent will convert the iodide  $I^-$  to iodine  $I_2$ .

### Literature References:

- Moffat, A.C., *Isolation and Identification of Drugs*, 2<sup>nd</sup> Edition, edited by E.G.C. Clarke, The Pharmaceutical Press: London, 1986, pp. 160-177, 1170-1171.
- Butler, W.P. "Methods of Analysis for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs", Internal Revenue Service Publication No. 341: Washington, D.C., 1967, pp.92-93.
- Moffat, A.C., Osselton, MD, and Widdop, B, *Clarke's Analysis of Drugs and Poisons*, 3<sup>rd</sup> Edition, Pharmaceutical Press: London, 2004.
- Gardner, Vickie, *Memorandum: Presumptive Tests to Identify "Bath Salts"*, FDLE Criminal Investigations and Forensic Science Program, February 1, 2011.

## 2.5 CRYSTAL TESTS

The following crystal tests are conducted as described below. For preparation of reagents, see Section 6 REAGENT PROCEDURES. Reagents are tested at the time of preparation to ensure that they are functioning properly, and the results are recorded in the Reagent Log Book. All reagents will be routinely re-tested every 6 months and the results recorded in the Reagent Log Book. See **Section 2.4** for Reagent Verification schedule.

Crystal tests are performed by placing a minute amount of the sample on a microscope slide (in some cases the sample must be dissolved in a solution such as dilute acid) and adding a drop of the reagent directly to it. A second technique is to slowly bring two solutions together with a glass rod or applicator stick. Crystals are then observed under a polarizing microscope.

### **Criteria for a POSITIVE response:**

- a. Test droplets that have dried may not be used for evaluation of crystals.
- b. Crystals must be compared to those of standards tested in the same manner.
- c. For frequently observed crystals, standards need not be run each time samples are run.

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- **Gold Chloride Crystal Test for Cocaine**

**Reagents:** 5%  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$  in distilled water  
20% Acetic Acid

**Procedure:** Place sample on a glass slide and add 1-2 drops acetic acid. Add one small drop of gold chloride solution and observe immediately on the polarizing microscope for the appearance of long rods with one or many arms at nearly right angles to the main axis (+’s).

**Reference:** Butler, W.P. “Methods of Analyses for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs”; Internal Revenue Service Publication No. 341: Washington, D.C., 1967.

- **Gold Chloride / Phosphoric Acid Crystal Test for Methamphetamine**

**Reagent:** 1 gram gold chloride ( $\text{HAuCl}_4$ ) dissolved in 20 milliliters 1:2 phosphoric acid ( $\text{H}_3\text{PO}_4$ ) in deionized water.

**Procedure:** Place sample on glass slide and add 1 or 2 drops of reagent. Observe formation of “plier” shaped crystals.

**Reference:** Aunan, Jayne. Washington State Patrol Laboratory, Spokane, WA.

- **Silver Nitrate / Cupric Nitrate Crystal Test for GHB**

**Reagent:** 100 mg of  $\text{AgNO}_3$  and 100 mg of  $\text{Cu}(\text{NO}_3)_2$  dissolved in 10 mL of water

**Procedure:** Place sample on glass slide and add 1 or 2 drops of reagent. Rectangular crystals grow at the edges in under 5 minutes. View crystals with a polarizing light microscope.

If suspect material is a solution, combine a drop of unknown with a drop of reagent via a connection made by an applicator stick.

**Reference:** Andera, Kevin, 93<sup>rd</sup> Semi-Annual CAC Seminar, Oakland, CA; May, 1999



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### 2.6 PROTOCOL for MARIJUANA and MARIJUANA PRODUCTS

The protocol for marijuana or Cannabis analysis is dependent upon the form submitted. When plant material is present, the identification will generally include both a botanical and a chemical identification.

The entire sample should be visually examined for homogeneity. Generally, marijuana products can be divided into:

- a. Plant material, crushed plant material, charred plant material, or seeds are all controlled as VIA if seeds are viable and charred material includes identifiable plant hairs (reference statute definition):

"marijuana" means the seeds, and leaves, buds, and flowers of the plant (genus) Cannabis, whether growing or not; it does not include the resin or oil extracted from any part of the plants, or any compound, manufacture, salt, derivative, mixture, or preparation from the resin or oil, including hashish, hashish oil, and natural or synthetic tetrahydrocannabinol; it does not include the stalks of the plant, fiber produced from the stalks, oil or cake made from the seeds of the plant, any other compound, manufacture, salt, derivative, mixture, or preparation of the stalks, fiber, oil or cake, or the sterilized seed of the plant which is incapable of germination;

- b. Residues, hashish, hash oil, and synthetically-produced cannabinoids (dronabinol or nabilone) are all controlled as IIIA (reference statute definition):

"hashish" means the dried, compressed, resinous product of the plant (genus) Cannabis;

"hashish oil" means the viscous liquid concentrate of tetrahydrocannabinols extracted from the plant (genus) Cannabis;

- **Macroscopic examination:**

Record a description of the item and packaging on the LIMS worksheet.

Indicate the results of the "**MORPHOLOGY**" examination. A **positive** result indicates that the identifying characteristics of marijuana are present, such as:

- a. palmate, serrated leaves
- b. flowering bud material
- c. seeds ovoid in shape
- d. stems are squared in cross-section

- **Weight:** If multiple units are present within an item, selection of samples for analysis should be based upon Alaska statute considerations. Charging for marijuana offenses is based upon 1 ounce and 4 ounce cutoffs, so analysts should be aware of these critical weights. (See APPENDIX II Weight Considerations in the Alaska Statutes.)

- **Stereomicroscopic examination:**



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For plant material, observe sample for hairs on leaf material. Marijuana leaves have three types of microscopic hairs on their surface. Cystolithic hairs are unicellular, contain deposits of calcium carbonate, and are shaped like bear claws. The opposite surface of the leaf will have long, thin, unicellular "clothing" or simple hairs. Mushroom-shaped glandular hairs may also be present. The presence of cystolithic hairs is the minimum requirement for a **positive** botanical identification.

For hashish preparations, a microscopic examination should distinguish between ground-up marijuana and true hashish, which is primarily plant hairs. Chloral Hydrate solution may be used to facilitate microscopic analysis for plant hairs. Identification of characteristic plant hairs combined with a chemical identification is sufficient for the identification of hashish.

Hash oil may be green or brown and will have no cellular material.

Note that a botanical identification is not usually possible with residue amounts.

Germination of seeds to demonstrate viability is not normally performed in this laboratory. Approval by the discipline supervisor or Laboratory Manager is required for this specialized testing.

- **Chemical Identification:**

Extract a portion of the plant material or swab of suspected substance with a suitable solvent:

- a. Borate buffer/dichloromethane
- b. Petroleum ether
- c. Methanol

If a residue only is being examined, two samplings should be obtained, extracted, and analyzed.

Analyze by GC/MS. The minimum criteria for *chemical* identification of marijuana or marijuana products (THC, hash, hash oil, Marinol™, etc.) is the presence of tetrahydrocannabinol ( $\Delta$ -9 THC).

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### 2.7 PROTOCOL for GENERAL DRUG (OR UNKNOWN) SUBSTANCES

The analysis of unknown specimens can be accomplished by a variety of methods. Limited size, the possibility of future analyses, and other limitations should be considered before testing is performed. The following is a generic protocol that outlines the analysis of a suspected drug sample. It should be noted that the nature of the sample determines the analytical route the forensic scientist pursues and that many samples do not lend themselves to an exact order of protocol. The forensic scientist ultimately decides the specific route for each sample. Analysts have discretion on which samples will be processed.

Review the Request for Laboratory Services form for latent fingerprint requests. If latent print work has been requested, special care in handling is required to prevent damage to any latent prints which may be present.

Remove the evidence from the packaging. If desired, photographic or digital imaging may be employed to document the original condition of the item. Visualization with a stereo microscope may aid in a description. Perform a preliminary examination of the item. If one substance is present, proceed to examine the item. If more than one "population" appears to be present, or multiple packages are present, follow Section 2.9 Sampling Protocols.

- a. Record a description of the item and packaging on the LIMS worksheet.
- b. Obtain a weight, count or volume of sample. See Section 2.3 Quantity Determination.
- c. If item is tablets/capsules, see Section 2.2 Literature Reference.
- d. Perform any applicable preliminary testing on sample. See Section 2.4 Color Tests and 2.5 Crystal Tests.
- e. Prepare sample for analysis. See Section 3.0 Extractions.
- f. Analyze the item with adherence to Section 1.4 Identification Requirements.
  1. For identification of any drug substance:
    - a. Two samplings utilizing the technical procedures in this manual are required.
    - b. At least one of the analyses must be GC/MS or FTIR.
  2. For physical identification only:
    - a. Literature search
    - b. Documentation in notes of hard copy source or copy of virtual source.
  3. For 'No Drugs per Alaska Statutes' it is required that a GC/MS analysis with a temperature program encompassing the range of the Quality Control Mixture be performed demonstrating a lack of controlled substances.

#### NOTE:

- The analyst may do more testing than the minimum required.

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- Unapproved tests, if used, may not be counted as part of a minimum analytic scheme.
- If quantity is insufficient for complete testing, the analyst will indicate **Not Analyzed** or **Quantity Insufficient for Analysis**.

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### 2.8 PROTOCOL for CLANDESTINE LABORATORY EVIDENCE

The objective in analyzing clandestine laboratory evidence is to determine if either a suspected lab site has the capacity to manufacture methamphetamine or has in fact done so. The ephedrine/pseudoephedrine reduction method is the method most commonly observed. Precursors and chemicals include:

- **Ephedrine or pseudoephedrine**

This precursor is normally extracted from over-the-counter tablets.

- **Iodine**

This reagent may be seen as crystals of  $I_2$  or as solutions of iodide,  $I^-$ . Rarely, labs may have access to HI (hydriodic acid), but the usual source of HI is the combination of iodine and any acid or iodine and phosphorus.

Iodine may be obtained from veterinary or medical sources, or as drug store "tinctures". Hydriodic acid is a colorless to dark yellow liquid obtained from chemical suppliers.

- **Phosphorus**

Red phosphorus is a reddish-brown powder. It may be obtained from chemical supply houses, road flares, fireworks, and matchbook striker plates.

The other type of ephedrine reduction utilizes lithium (or sodium) metal and anhydrous ammonia:

- **Lithium**

Lithium is extracted from lithium batteries with tin snips or obtained commercially. It is usually stored in toluene or mineral oil because of its explosive, reactive nature with water. *Lithium is not analyzed in this laboratory.*

OR

- **Sodium**

Obtained from a chemical supply house or made by electrolytic deposition, using sodium hydroxide, copper tubing, and a car battery. Sodium metal reacts violently with water, causing fire and explosion unless kept under kerosene. *Sodium is not analyzed in this laboratory.*

- **Pseudoephedrine or Ephedrine**

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- **Anhydrous Ammonia gas**

This may be found stored in pressurized cylinders or propane tanks, often in agricultural settings (as fertilizer), fish-processing plants, and ice-rinks. Gas is released into a thermos bottle as a liquid. *Ammonia is not analyzed in this laboratory.*

Other chemicals may be submitted as evidence but are not typically analyzed:

- **Acids** (Hydrochloric or Muriatic, Sulfuric)
- **Solvents** (Toluene, Coleman fuel, Acetone)
- **Alcohols** (for pill extraction)
- **Drying Agents** (Epsom salt)
- **Filters** with Residues
- **Tubing** from HCl gas generators
- **NaOH**, usually as a liquid drain cleaner

Filters or tubing with drug residue may sometimes be analyzed for finished product.

- **SPECIMEN HANDLING**

Approach all clandestine laboratory specimens with extreme caution. Always use gloves and beware of inhaling caustic fumes. Due to the hazardous nature of many of the chemicals used, consideration should be given to opening all evidence in a fume hood. Leaking samples should be repackaged and samples should be re-sealed with a heat sealer if possible.

- **ANALYSIS**

### **A. DRUGS by GC/MS**

Extracted solids and liquids can be analyzed by GC/MS. Ephedrine reduction by-products, such as P2P, naphthalenes, aziridines, and oxazolidines can be identified by GC/MS library searches but are not included in the report. Drugs extracted from confiscated liquids must have a second sampling analysis.

### **B. PHOSPHORUS by GC/MS**

Phosphorus can be identified by GC/MS after converting red phosphorus to white phosphorus.

A small amount (1-2 mg) of red/brown powder substance is added to a test tube. The tube is then heated in the flame of a propane torch. Perform this procedure under a

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fume hood and always wear safety goggles! As the red phosphorus burns, white phosphorus begins to accumulate on the sides of the test tube. Remove the tube from the flame after 10-15 seconds and add 1-2 milliliters of dichloromethane. Be extremely careful of flash back at this point, as the dichloromethane is volatile and the phosphorus is explosive. Transfer the dichloromethane/white phosphorus mixture to an auto sampler vial.

Inject the sample on the GC/MS. Once the run is complete, look for a peak with the ions 31 (P1), 62 (P2), 93 (P3), and 124 (P4). Compare unknown spectra to that of a phosphorus standard prepared in the same way as the unknown sample.

### C. IODINE

Iodides may be identified by means of the well-known color reaction in which free  $I_2$  gives a blue color with starch. See **Section 2.4 Color Tests**.

Reference: Chamot and Mason, Handbook of Chemical Microscopy, Volume II

Distillation in the well of a spot plate (or in a test tube) is a means of separating the liberated  $I_2$  from substances that might interfere with the "starch-iodide" test. A small amount of the substance to be tested is placed in the well of a spot plate or a test tube. Several drops of dilute acid containing an oxidizing agent may be added to oxidize any  $I^-$  to  $I_2$ . Cautious heating aids in volatilizing the  $I_2$ . A few granules of starch placed on a microscope slide or a cotton ball are placed over the well or in the test tube respectively. A blue color indicates the presence of iodine.

Sublimation can also be considered as indicative of iodine. Blue-black iodine crystals can convert directly from solid form to a violet-colored gas phase.

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## 2.9 SAMPLING PROTOCOLS

The following guidelines dictate the laboratory's policies on sampling. Conclusions reported must clearly state to the user of the report exactly what was analyzed to reach the stated conclusions.

- General sampling guidelines:
  1. Visually different samples within an item will be separated for analysis.
  2. Visually different samples within an item with potential cross contamination may not need separate analyses.
  3. The analyst should leave a portion of the original sample for potential re-analysis.
  4. A minimum of two samplings shall be separated for analysis from each item to be tested (one presumptive and one confirmatory).
- Single sample items:
  1. If the sample appears homogeneous, a portion is simply removed.
  2. If an item is not homogeneous (for instance, cocaine salt mixed with cocaine base) or is very large, multiple sampling can be done. The worksheet and report should reflect the mixture of drug materials.
- Multiple sample items  
(Before employing sampling protocols, be familiar with Section 2.1 Visual Screening and Appendix II Weight Considerations in the Alaska Statutes):
  1. If the gross weight of the entire population is above a critical weight and 20 or less samples from that population are required to exceed that critical weight, full analysis will be performed on that number of samples regardless of population size.
  2. If the gross weight of the entire population is above a critical weight and more than 20 samples from that population are required to exceed that critical weight, a hypergeometric sampling plan (90% positive with a 95% confidence interval) will be used to determine the number of samples to be analyzed (see Appendix IV Hypergeometric Sampling Plan). If any samples differ from the rest, the analyst will reevaluate the item population.
  3. If the gross weight of the entire population is below the minimum critical weight or there are no critical weights for the drug in question, only one sample may be analyzed.

Example A:

55 tablets, all appear visually the same with pharmaceutical markings

1. Record description of packaging and tablets on worksheet.
2. Gross weight (or count) of all tablets is recorded on worksheet.



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3. Literature search (unless clandestine)
  - If controlled, perform GC/MS of one tablet
  - (If not controlled, report drug and “physical identification only”)
4. **DESCRIPTION:** Blue tablets  
55 count  
1 tablet analyzed
5. **RESULTS:** Oxycodone, 1 tablet

Example B:

- 8 baggies of plant material, all appear visually the same
1. Record description of packaging and contents.
  2. Perform a gross weight of all 8 baggies.
  3. If the total weight apparently exceeds a threshold amount of 1 ounce or 4 ounces, perform a botanical identification and GC/MS on as many baggies as needed to reach the threshold amount.
  4. If the total gross weight is less than 1 ounce (28 grams), perform a botanical identification and GC/MS on the contents of *one* baggie & repackage.
  5. **DESCRIPTION:** 8 baggies Plant Material  
Gross weight, xx.x grams  
Contents of 1 baggie analyzed
  6. **RESULTS:** Marijuana, x.x grams

- **NOTE:** At an analyst's discretion, additional presumptive or confirmatory testing may be performed beyond the sampling plan policy.

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### 2.10 EXTRACTIONS

Many drug samples are mixtures or contain excipient material requiring the compound of interest to be separated from a matrix before subjecting the sample to further instrumental analysis. Information as to solubilities and specific physical properties can usually be found in Clarke's Isolation and Identification of Drugs. The choice of an organic solvent is dependent upon the drug to be extracted and the preferences of the analyst.

- Solvent extraction - the analyte is dissolved in an appropriate solvent and separated away from insoluble excipients. The solvent is then analyzed.
- Solvent washes - the excipient material is dissolved and washed away from the analyte by using a solvent in which the analyte is insoluble.
- Solvent/solvent extractions - two immiscible solvents can be used to extract an analyte. An aqueous phase (acidic or basic) and organic solvent phase are typically used. Many drugs are nitrogenous compounds that readily convert between salt and free species enabling them to be separated by acid/base and organic solvents.
- Particle-picking - some mixtures and crystalline samples can be physically separated and isolated. A stereomicroscope can aid in the isolation of individual components of a sample.

A more comprehensive outline of extraction options follows:

#### A. Physical Separation

Manually separate material by physical appearance. This includes scraping or shaking material from currency, paraphernalia, clothing, etc.

#### B. Dry Extraction

1. Place a portion of the sample in a disposable test tube.
2. Add a solvent that dissolves the drug of interest, but not excipients.
3. Decant or filter to separate the solvent and discard any insoluble material.

#### C. Collection of Residues

Paraphernalia may be rinsed by pouring solvent onto/through the item OR swabbing with a cotton-tipped applicator moistened with water or solvent.

#### D. Liquid/liquid extraction

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### 1. Basic extraction

- a. Dissolve the sample in ~ 5% borate buffer (or other suitable basic solution).
- b. Add dichloromethane (or other appropriate solvent).
- c. Vortex or shake.
- d. Resulting layers contain:
  - i. Aqueous Layer – Acidic chemicals.
  - ii. Organic Layer – Basic and neutral chemicals.

### 2. Acid extraction

- a. Dissolve the sample in 0.1N H<sub>2</sub>SO<sub>4</sub> (or other suitable acidic solution).
- b. Add dichloromethane (or other appropriate solvent).
- c. Vortex or shake.
- d. Resulting layers contain:
  - i. Aqueous Layer – Basic chemicals.
  - ii. Organic Layer – Acidic and neutral chemicals.

### 3. Acid/base extractions (Back Extractions)

- a. Dissolve sample as described in 2a.
- b. Add organic solvent as described in 2b.
- c. Shake and separate layers.
- d. Resulting layers contain:
  - i. Acidic Aqueous Layer - Basic chemicals.
    - a. Adjust pH of aqueous layer to ~ 9-14 using base.
    - b. Add dichloromethane.
    - c. Shake.
    - d. Resulting layers contain:
      1. Basic Aqueous Layer - Organic insolubles.
      2. Basic Organic Layer - Basic chemicals
  - ii. Acidic Organic Layer - Acidic chemicals.

### E. Gammabutyrolactone (GBL) or 1,4-Butanediol (BD) Extraction:

1. Combine approximately equal volumes of the sample liquid and dichloromethane in a test tube. (2 – 3 milliliters of each when sample size permits). If the sample is powder, take up in water, and then extract with dichloromethane.
2. Vortex or shake.
3. Let settle.
4. GBL/BD will be in the dichloromethane layer. If Gammahydroxybutyric acid (GHB) is present, it will be in the aqueous layer. Remove dichloromethane and analyze by GC/MS.

### F. Gammahydroxybutyric Acid (GHB) Procedure

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Perform a microcrystalline test and then if positive proceed with the following protocol:

1. Remove a portion of sample (or take the aqueous layer from "E" above) and evaporate to dryness. Keep dried sample at ~105° C or place in a dessicator.
2. GHB Derivatization Procedure using N-methyl-N-trimethylsilyltrifluoroacetamide (MSTFA)
  - a. Place MSTFA in three auto-sampler vials or auto-sampler vials with glass inserts.
  - b. Add a couple of mg of unknown sample to the first vial. To the second vial add a couple of mg of the Standard GHB. Leave a third vial blank with only MSTFA.
  - c. Derivatize capped vials at 90° C for 10 minutes (hotplate or warm water bath).

### G. Mushroom Extraction Procedure

1. Extraction Procedure 1
  - a. Grind mushrooms to a fine powder.
  - b. Soak 1 to 2 grams in ethanol for 30 minutes, vortexing every 15 minutes for 30 seconds.
  - c. Add 10 drops of 20% acetic acid.
  - d. Soak an additional 30 minutes with vortexing for 30 seconds.
  - e. Centrifuge sediment to bottom and transfer acidic ethanol to a test tube.
  - f. Evaporate to dryness.
  - g. Reconstitute with dichloromethane, vortex, and analyze by GC/MS.

### H. Clorazepate Extractions

1. Extraction Method 1
  - a. Place powder from a capsule or crushed tablet in a container.
  - b. Add approximately 3 milliliters of 15N ammonium hydroxide.
  - c. Stir the mixture.
  - d. Centrifuge or allow to settle.
  - e. Remove the aqueous layer and evaporate. The resulting residue is the clorazepate salt, suitable for FTIR analysis.
2. Extraction Method 2
  - a. Place powder from a capsule or crushed tablet in a container.
  - b. Add approximately 12-15 milliliters of dichloromethane and methanol mixed in a 3:1 ratio.
  - c. Mix vigorously.
  - d. Centrifuge or allow to settle.
  - e. Remove the liquid and filter it through a filter presoaked with the dichloromethane:methanol mixture.
  - f. Add 1.5 to 2 milliliters of water to the filtrate and mix thoroughly.

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- g. Centrifuge or allow to settle.
- h. Remove the aqueous top layer and wash it twice with dichloromethane, centrifuging after each wash. (If the dichloromethane layer contains a white opaque foam-like substance after the second wash, repeat the wash until the dichloromethane layer is clear.)
- i. Evaporate the aqueous layer. The resulting residue is the clorazepate salt.

### LITERATURE REFERENCES

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### 2.11 NEW METHOD VALIDATION

This procedure will apply to new method validations used by the analysts in the controlled substances discipline. Actual validation protocols will vary, depending upon whether the method is laboratory-developed or has been already validated at another certified laboratory.

- **GENERAL**

- A. Perform a literature search in the area of interest.
- B. Develop specific objectives for the method. Include reasons why the procedure may be of value. Discuss development of the method with supervisor and other discipline members.
- C. Plan a series of steps required to perform the experiment (methodology).
- D. Perform the experiment. Note any changes made to the initial procedure.
- E. Prepare a validation/verification report documenting the study. Subjects addressed in the report should include:
  1. Objective
  2. Theory/background
  3. Chemicals/reagents/equipment
  4. Instruments/parameters
  5. Methodology/procedure
  6. Summary of data/results  
Spectra/chromatographs/tables/graphs
  7. Discussion of results  
Applicability  
Limitations  
Specificity  
Reproducibility  
Usefulness
  8. Bibliography of references and cited works
- F. Submit report to the Controlled Substances Supervisor for approval.

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## SECTION 3 INSTRUMENTAL PROCEDURES and QUALITY CONTROL

### 3.1 GAS CHROMATOGRAPHY / MASS SPECTROMETRY

The State of Alaska Scientific Crime Detection Laboratory utilizes three Agilent GC/MS instruments for analysis of drug substances. Additionally, a fourth GC/MS utilized for fire debris evidence can be utilized for separation of co-eluting drug peaks because of its different column. Information specific to the GC/MS used for fire debris can be found in the Fire Debris Manual. Information regarding all instrument specifications is kept:

1. In individual instrument **logbooks** kept with the instruments in the Instrument Room. This includes specific methods and operating parameters.
2. Laboratory computer network: [I:\Discipline Shares\Controlled Substances\Instrumentation](#)
3. In the LIMS under **CS INST 2011** (or current year):
  - a. Unique identifier (Curly, Abbott, Larry, or Moe)
  - b. Serial numbers
  - c. Software version
  - d. Manufacturer
  - e. Location, and
4. Through the Agilent **HELP** function, which accesses the electronic instrument manual and gives specific instructions on the use of the GC/MS and Chemstation software, and
5. Agilent's electronic hardware manual for GC/MSD.

GC/MS has the advantage of giving two useable identification criteria from one sampling, and supplying information concerning molecular structure. Limiting considerations include:

- a. Not all samples are amenable to GC/MS analysis. Thermally labile and non-volatile samples are not readily analyzed.
- b. Complete destruction occurs with the injected portion of evidence.
- c. Spectra from closely related compounds and isomers may be so similar that conclusive identification is not possible.

- **Procedure for GC/MS analysis:**

- A. Prepare reagent blanks or solvent blanks to precede each casework sample being analyzed. Run a blank before each evidence sample using the same instrument conditions (oven temperature program, split ratio, injection volume, & multiplier voltage).
- B. Prepare sample solutions according to **Section 2.10 Extractions** and transfer to autosampler vials.
- C. Make sure the liquid level is above the syringe injection depth.



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- D. A temperature program encompassing the range of the Quality Assurance Mixture must be used to assure resolution of drug components.
- E. Sample injection shall be by autosampler.
- F. The following information should appear on the printout header:
  - a. The GC/MS instrument (Larry, Curly, or Abbott).
  - b. Acquisition program utilized (Screening). Details of the program method are referenced in each instrument's Log Book.
  - c. Laboratory case number
  - d. Item number.
  - e. Analyst (If another analyst is logged on to the instrument, add initials to header in comment section.)
  - f. Date and time of sample injection
  - g. Vial number
- G. A standard must be referenced for each drug substance reported. If the laboratory has the drug standard in its inventory, analysis of the standard within one month of the unknown must be included in the case file. The printout of the total ion chromatogram and spectra from that standard will include the lot number (unique identifier). This lot number can be used to track the quality control information stored in the Drug Standards QC binder (see **Section 5 Reagents and Standards**). Note the exception (see page 4) for therapeutic analgesics in a pharmaceutical preparation including an opioid drug.
- H. If the laboratory *lacks* the drug standard, EITHER documentation of a library search with an approved spectral library OR comparison to documented and approved spectra that has been scanned into the case file must be referenced. Two samplings are still required to meet identification criteria
- I. All autosampler runs will be checked for proper sequencing prior to starting and once the run is complete. This does not need to be documented.
- J. When instrumental acquisitions are complete, the analyst will review the data and put a copy into the LIMS case file. Each electronic and/or hard copy will include the header information listed above, a total ion chromatogram (if generated from an in-house standard), and the mass spectrum of each peak of interest. After all reviews are complete and a report has been distributed, any hard copies are destroyed. The electronic notes and report in LIMS should be the only copy of the case file (with the exception for court testimony).
- K. Since data interpretation is done from the electronically generated files stored in the case files, this is the only instrumental data that must be retained. All raw data may be deleted and retention of these files is at the analyst's discretion.

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- **Interpretation of Data and Criteria for Identification by GC/MS:**

A. Reagent or solvent blanks were run before all casework samples, utilizing the same instrumental conditions as the sample. Blanks must not contain peaks of interest. Documentation of the blank samples is retained in the case file.

B. All data (retention time and spectra) from analyzed controlled substances will be compared to spectra from verified in-house drug standards, when available, within a one month time frame utilizing the same temperature program. Documentation of these standards is retained in the case file. If a chemical standard is not available, an identification can be made on the basis of mass spectra only, utilizing approved spectral libraries or approved outside sources. The requirement for two samplings is not waived. See Section D below for guidelines on interpretation of mass spectra.

C. Comparison of retention time data between evidence samples and primary standards must demonstrate agreement within  $\pm 5\%$  or 0.05 minutes (3 seconds).

D. The mass spectrum of each evidence sample is visually compared with that of a primary standard. The significance of peaks (both absent and present) is noted and no prominent ions should be missing from the evidence spectrum. For a match to be considered acceptable, the main ions should agree between unknown and standard and the presence or absence of a 'molecular ion' must agree between unknown and standard. Unknown sample spectra may contain additional peaks due to background or sample impurities. If the spectra from an evidence sample is lacking in detail and the analyst feels that it is too weak to call, the analyst may report "insufficient concentration for identification". This will indicate that the analyst *did* perform an analysis, sufficient data existed to *suggest* the presence of a specific substance, but the results did not meet the criteria for identification. The criteria for the acceptance of a spectral match between unknown evidence and a known standard is the same whether the standard is from a reputable library or analyzed in-house from the laboratory's drug inventory.

Disagreements between an analyst and technical reviewer regarding the interpretation of spectra should be a source of discussion within the discipline so that all analysts are in agreement with 'calling criteria'.

**NOTE:** When there is disagreement concerning *instrumental* 'calling criteria' for blanks, retention time, or mass spectrum (e.g. carryover in a blank sample, molecular ion in standard but not evidence), the analyst must repeat that portion of the analysis and resolve the discrepancy before reporting results. If resolution by the analyst and technical reviewer cannot occur, the Controlled Substances Supervisor or designee will decide the issue.

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- **Uncertainty of Measurement of GC/MS and Good Lab Practices**

The biggest uncertainty in detection and measurement when utilizing GC/MS technology is contamination prevention. The second potential source of error is in mislabeling or misplacement. To assure that contamination or mishandling of evidence does not occur, the laboratory adheres to the following practices:

- A. Only one item of evidence should be analyzed at a time.
- B. Use disposable gloves, bench paper, pipets, wipes, test tubes, weigh papers, and vials when handling evidence.
- C. Reusable items (spatulas, forceps, beakers, scissors, etc.) must be cleaned prior to each use.
- D. Balances should be cleaned as needed and used with disposable trays.
- E. Batch processes (autosampling) require that each item be labeled.
- F. A reagent blank or solvent blank should be prepared and run on the GC/MS prior to each case sample. Documentation of this is maintained in the case file.
- G. If an extract is to be concentrated using the water bath evaporation system, the probes are cleaned prior to use.
- H. Placement of autosampler vials is checked before starting sequence.

- **Safety**

- A. Take proper precautions when moving compressed gas cylinders.
- B. Follow instrument manual operating instructions.
- C. Follow proper safety precautions when operating and maintaining the GC/MS instruments as per instrument manuals.

- **Maintenance of GC/MS instruments**

The following protocols are established as a preventive maintenance program for gas chromatograph/mass spectrometers. All maintenance, both preventive and corrective, must be described and dated when recorded in the instrument logbooks. Exceptions to this include routine computer re-booting and exchange of helium tanks.

**Agilent MSD GC/MS** – The Department of Public Safety maintains service contracts with Agilent for technical support, service, and parts. At this time, the service contracts do not include any routine preventative maintenance (source cleaning, pump oil changes, adjustments, lubrication, inspections, or column changes), so these actions are normally performed by crime lab personnel on site. On occasion, analysts may be called upon to install filaments, a voltage multiplier, or other replacement parts.

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**Instrument Computer Interface** – All GC/MS instruments are interfaced with computers approved by Agilent. All computers are maintained according to Department of Public Safety Information Technology protocols.

- **Columns** - Columns that are installed in the Gas Chromatograph/Mass Spectrometer shall have an approximate length of 30 meters and have a 5% phenyl/95% polymethyl silicone phase, 100% methyl silicone, or an alternate column as approved by the discipline supervisor.

Maintenance cuts to the columns to enhance column performance are permitted. These columns will be used to run the QC mix and for general drug analysis. Columns are replaced as needed.

**Compressed helium** – used as a carrier gas. Tanks are stored in the building and the gas is transported to the instruments in metal tubing. A manifold system allows rapid change from one tank to another when the gas is nearing depletion. New tanks of helium are obtained from Air Liquide, an ISO 17025 certified supplier. Installation of new tanks may be performed by the laboratory's maintenance specialist or anyone in the controlled substances discipline of the laboratory.

**Hardware consumables** – Septa, glass inlet liners, and autosampler syringes are replaced as needed.

- **Quality Control of GC/MS:**

**Perfluorotributylamine (PFTBA)** – calibration standard. The GC/MS instruments are calibrated weekly (when in use) utilizing the Autotune program. Reports of calibrations (Autotune reports) are electronically stored in the Laboratory Information Management System (LIMS) in a file designated **CS INST 2011** (or current year). To access the files in LIMS, click on the camera and open "Case Images". Note folders and sub-folders within the image section.

A successful calibration is indicated when the instrument is able to assign proper masses to the PFTBA fragments. Critical components are:

- A. Passing the System Verification in the Tune Evaluation program
- B. Peaks are symmetrical in shape.
- C. Mass 69 is the base ion.
- D. Water and air abundances pass the Tune Evaluation (are less than 20% of the 69 abundance).

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**Quality Control (QC) Mix** – a mixture of four 1 mg/mL drug substances specifically selected to demonstrate the retention time range for controlled substances in Alaska and definitive peak resolution between closely related drugs :

- A. Dimethyl Sulfone (MSM)**
- B. Tetracaine**
- C. Cocaine**
- D. Buprenorphine**

The QC Mix is analyzed weekly on each instrument. Each GC/MS must identify each of the four components with acceptable **GC** resolution, sensitivity, and peak shape on the total ion current chromatogram (TIC) and acceptable **mass spectra** for the cocaine peak.

A successful performance check is indicated by:

- A.** Detection of the four components of the QC Mixture.
  - B.** Baseline resolution between tetracaine and cocaine.
  - C.** Symmetrical peaks with minimal tailing.
  - D.** The cocaine peak of the QC Mix giving an appropriately detailed mass spectrum when compared to a reference standard spectrum.
- Interpretation of mass spectra is the same with the QC Mix as with casework (see **Interpretation of Data and Criteria for Identification by GC/MS**) on page 31. To conform to this requirement, the spectra should include the following ions:

Cocaine	m/z 303
---------	---------

Each GC/MS must pass QC check (autotune calibration and QC Mix control) each week before it can be used in casework. Any maintenance such as a column change or hardware replacement requires a QC check before casework can be resumed with that instrument. An autotune is recommended after liner and septum changes but not required.

**QC Evaluation:** If any of the criteria above are not met, then the instrument does not pass the quality control check and must not be used in casework. The instrument must undergo further maintenance and/or troubleshooting to correct any problems and a subsequent quality control check must be performed. Entry of Autotune, Tune Evaluation, and QC Mix data into the electronic data storage of Justice Trax is made when the data satisfies all requirements.

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- **Records:**

The following documents must be maintained in the laboratory for each GC/MS. Some data may be duplicated between hard copy in the logbooks and electronic storage in LIMS:

- A. A description of the instrument system
- B. Documentation of weekly calibration (Autotune) data.
- C. Documentation of weekly QC Mix data.
- D. Documentation of all maintenance and repairs.

- **Bibliography**

Saferstein, R., Ed. *Forensic Science Handbook*; Prentice-Hall: Englewood Cliffs, NJ, 1988, Vol.III.

Yinon, J., Ed. *Forensic Applications of Mass Spectrometry*; CRC: Boca Raton, FL, 1987.

McLafferty, F.W. *Interpretation of Mass Spectra*, 4<sup>th</sup> Edition; University Science: Mill Valley, CA, 1990.

Skoog, D.A. *Principles of Instrumental Analysis*, 3<sup>rd</sup> Edition; Saunders College Publishing: New York, 1985, pp. 523-535, 554.



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### 3.2 INFRARED SPECTROPHOTOMETRY (FTIR)

The State of Alaska Scientific Crime Detection Laboratory utilizes a Thermo Nicolet iS10 Infrared FTIR Spectrometer with an ATR (attenuated total reflectance) accessory for analysis of drug substances. Information regarding the instrument specifications is kept in the instrument logbook and manuals kept with the instrument in the Instrument Room. Information on the infrared spectrometer is also stored on the Lab's computer network: <I:\Discipline Shares\Controlled Substances\Instrumentation\FTIR>.

Infrared analysis is non-destructive and sample size is small. However, the sample must be relatively pure for conclusive identification. Inorganic compounds often lack the spectral complexity necessary for identification.

- **Procedures for FTIR analysis:**

- A. If significant amounts of interfering substances are present, extract the sample using any extraction which successfully isolates the substance of interest. Use caution to prevent conversion between base and salt forms.
- B. Prepare the sample for analysis by one of the following techniques:

1. **ATR (Attenuated Total Reflectance)**
  - a. Place small amount of sample on ATR crystal.
2. **Potassium bromide (KBr) pellet**
  - a. Grind sample with KBr in a mortar using a pestle.
  - b. Use the die assembly and press to prepare a pellet.
3. **Neat liquid samples** on potassium bromide pellets
  - a. Prepare two Potassium bromide disks.
  - b. Place small drop of sample between disks.
4. **Salt plates**
  - a. Place a drop of liquid on one plate.
  - b. Press plates together by hand.

- C. Acquire a background spectrum (blank) of air. The blank must be analyzed using the same instrumental conditions as the sample and retained in the case file.
- D. Perform a quality control check by acquiring an absorbance spectrum using a 1.5 mil film of polystyrene. See **Quality Control of FTIR**.
- E. Acquire sample absorbance spectrum and search libraries for an identification.



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- **Interpretation of Data and Criteria for Identification by FTIR analysis:**

- A. For identifying a reportable drug, the sample spectrum must be visually compared with the spectrum of a standard, either run on the same instrument using the same sampling mode (absorbance, ATR), or the sample spectrum must be visually compared to a library-generated spectrum.
- B. Unknown materials may contain extra absorbance bands due to sample impurities. The significance of absorbance band peaks (both absence and presence) and relative intensities of absorbance bands should be assessed. However, no prominent bands should be missing from the unknown spectrum.

- **Maintenance**

- A. Record all maintenance in the instrument logbook kept beside the FTIR in the instrument room.
- B. Routine maintenance performed as needed:
  - 1. Dessicant replacement
  - 2. Inspection of humidity indicator
  - 3. Cleaning of mirrors (factory service only)
  - 4. Alignment of spectrometer
  - 5. Cleaning of spectrometer, accessories, computer, monitor, and keyboard

- **Quality Control**

- A. Perform a background acquisition on all days that the instrument is used.
- B. Acquire spectra from a polystyrene standard. Check for absorption at  $1601\text{ cm}^{-1}$ ,  $\pm 2\text{ cm}^{-1}$ . This peak can be labeled using the 'T' function in the annotate toolbar. The polystyrene spectra should give a correlation match of 90 or greater with library spectra. Record the polystyrene results in the FTIR computer notebook and insert the spectra into the LIMS file.
- C. Instrument validations are run periodically and after any major service or hardware replacement. The manufacturer's software program shall be used to determine performance of wavenumber accuracy, resolution, and signal-to-noise. The field on these reports for annotating the scientist performing the test, date, and verification by another scientist need not be filled out since this information is satisfied by the name and date appearing on the document.
- D. Record all validations (performance verifications) in the LIMS in the folder CS INST 2011 or 2012.

- **Records**

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The following documents must be maintained in the laboratory for the Infrared Spectrometer. Some data may be duplicated between hard copy in the logbook and electronic storage in LIMS:

- A. A description of the instrument system
  - B. Documentation of polystyrene performance data
  - C. Documentation of validation checks
  - D. Documentation of all maintenance and repairs
- **Bibliography**

Smith, B.C. *Fundamentals of Fourier Transform Infrared Spectroscopy*; CRC Press: Florida, 1996.

Karasiewski, R. *Basic Training Program for Forensic Drug Chemists*; 3<sup>rd</sup> Edition, Drug Enforcement Administration, 1998, Chapter 5, pp. 29-48.

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### 3.3 BALANCES

The State of Alaska Scientific Crime Detection Laboratory utilizes four Mettler Toledo balances for weighing of drug substances. Information regarding the balance specifications is kept in the Balances Logbook and the manuals stored in the Controlled Substances laboratory. Additionally, this information is stored on the Lab's computer network: [I:\Discipline Shares\Controlled Substances\Instrumentation\Balances](#) and in the LIMS folder CS INST 2011 (or 2012).

- **Procedure**

See **Section 2.3 Quantity Determination**

- **Maintenance**

- A. Make sure the balance is level and protected from vibration.
- B. Keep all balances clean and dust-free.

- **Quality Control**

- A. All balances will be checked with NIST traceable standard weights on a monthly basis. Reference weights must not be touched with bare hands (tweezers or gloves are used).

Wyatt, Morgan, & Virgil:	0.50 grams	±0.01 gram
	10.00 grams	±0.01 gram
	100.00 grams	±0.01 gram
Wilbur:	0.5 grams	±0.1 grams
	10.0 grams	±0.1 grams
	100.0 grams	±0.1 gram
	4000.0 grams	±0.1 gram
- B. When a balance is physically moved, it must be re-verified with the standard weights.
- C. All balance checks will be documented in the balance logbook.
- D. All new balances will be checked with NIST traceable weights prior to use in casework.
- E. All balances will be calibrated and serviced on an annual basis by a vendor utilizing NIST traceable weights. The calibration will be recorded in the balances logbook and/or maintained in the Quality Assurance Manager's records.
- F. A repaired balance must be re-calibrated prior to use.

- **Uncertainty of Measurement Budget**

- A. Operator Uncertainty – All personnel using balances in the testing of controlled substances are by training and experience fully qualified in the proper use of the equipment.

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- B. Environmental Conditions – All balances are operated in a laboratory environment consistent with manufacturer's recommendations. Calibration checks and calibrations are performed at the locations where the balances are used.
- C. Calibration Procedures – All calibrations are performed in accordance with manufacturer's recommendations. Balances are checked monthly and re-calibrated if necessary. Annual calibrations are performed by an accredited vendor.
- D. Weight (Mass) Standards – Proper handling procedures for these standards are documented in **Section 2.3 Quantity Determination**, **Section 3.3 Maintenance**, and **Section 3.3 Quality Control**. The standards have been certified as NIST traceable by the supplier (VWR/SP) and are re-certified on an annual basis by an accredited vendor. NIST certification documents are kept in the balance logbook and on the Laboratory computer network:  
<I:\Discipline Shares\Controlled Substances\Instrumentation\Balances>
- E. Instrument Performance – Instrument performance is checked monthly using NIST traceable mass standards. Measurements obtained are for a range of weights appropriate for each balance and relevant to laboratory needs. These measurements may potentially be used to calculate standard deviation values.
- F. Sampling – Since *net* weights of evidence are usually obtained, residual sample may be left in an evidence container (bindle, pipe, baggie, etc.). This difference in weight is not significant and will always result in a lower reported weight than what is actually present.
- G. Truncating – Policy requires that weight quantities be truncated. As with sampling (see above), any discrepancy is a lower reported weight than the actual weight.
- H. Moisture Content of Evidence – It is beyond the control of the laboratory to dictate the moisture content in submitted evidence. This can then potentially result in a change of evidence weight over time as the substance either loses or gains weight with the drying or absorbing of water from ambient air.

- **Records**

The following documents must be maintained in the laboratory for the balances. Some data may be duplicated between hard copy in the Balances Logbook, hard copy kept with the Administrative Assistant and the Quality Assurance Manager, and electronic copies stored on the laboratory computer network:

- A. Descriptions of all balances with locations and serial numbers
- B. Documentation of monthly calibration checks
- C. Documentation of weight and balance validations
- D. Documentation of all maintenance and repairs

- **Bibliography**

- A. Balance manuals
- B. Balance logbook

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### 3.4 INSTRUMENT INTERFACES (COMPUTERS)

Computers used to interface instrumentation in the laboratory shall be supplied by or approved by the manufacturer of the instrumentation.

- **Maintenance**

- A. Restart computers weekly (at a minimum) to activate upgrades and patches.
- B. Install LIMS upgrades at the direction of the Lab Manager/LIMS Administrator.

### 3.5 NEW INSTRUMENT VERIFICATION PROCEDURE

- **GC/MS** At this time, all installation and verification of new MSD's is done by an approved factory representative. The documentation of this process is kept in each instrument's log book and includes a checkout tune, tune evaluation, and sensitivity check. Also checked are PFTBA peak shapes, mass assignments, and mass axis stability. A signal to noise report and tune report are generated by the technician.
- **FTIR** New instruments or attachments to existing instruments will be verified by the factory representative and documentation provided to the Chemistry Supervisor or his/her designee.  
  
Additionally, an in-house validation will be performed to demonstrate that the new instrumentation is equal or superior in performance to the replaced instrument.

A memo from the Controlled Substances Supervisor to the QA Manager documents a new instrument going into service, includes the serial number, and outlines the basis of the verification process. Paperwork with the memo includes the hardware and software installation checklist. All documentation included with the memo should ensure that the instrument was correctly installed and functioning as designed.

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## SECTION 4 ADMINISTRATIVE PROCEDURES

### 4.1 CASE FILE PROCEDURE

- **Bench Notes (Worksheet)**

The administrative aspects of casework analysis are found primarily within LIMS under the **Requests** for Analysis tab. It is here that the request is created, pertinent information added, prioritizations conveyed, evidence is requested, bench notes are recorded, reports are written, reviews are recorded, and distribution of the report is documented.

Proper note-taking is essential from the time the evidence is received by the analyst until returned to the Evidence department. Notes need to be complete and understandable by another discipline analyst, as they may be referred to months or years after the analysis was performed.

Proper notes should include the following information on the WORKSHEET:

- A. Any inconsistency between the description on the submission form and the evidence received.
- B. Dates that the analyst began analysis and date completed. To imbed this information in the worksheet, go to **Edit Request** and enter the two dates in the Assignor block, using the format MM/DD/YYYY.
- C. Description of each item's packaging. Note any packaging problems or unusual circumstances.
- D. Physical description of the evidence:
  - Type – powder, liquid, plant material, tablet, etc.
  - Color
- E. Sampling protocol employed.
- F. Weight or count of item. If multiple units are present within an item, obtain a gross weight of the entire item before sampling protocols are employed. Record this weight on the worksheet.
- G. Sample preparations or extraction methods used.
- H. Description of each analysis and the result.
- I. Conclusions upon completion of analyses.

And the following instrumental and reference data in the IMAGES section:

- A. Chromatograms and spectra used to reach conclusions.

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- B. Reagent blanks.
- C. Standards.
- D. Literature references or monographs if needed. (Add lab case number and analyst's initials and scan to create a pdf file.)

All case file documentation is to be sequentially numbered and the total number of pages conveyed. Note that this numbering is generated when an "e-case file" is created in the LIMS.

- **Case Images**

This portion of the case file in LIMS contains all Requests for Laboratory Services (RLS's) and subpoenas associated with the case, as well as any additional case information submitted by the law enforcement agency or Department of Law. Items not already in electronic form that need to be saved with the file can be scanned and added to the LIMS images.

- **Communications**

An acceptable form of documenting communications concerning a case is to use the Case Activities function (under the Case Info tab) in the LIMS database.

Inquiries concerning specific aspects of a case such as triaging may be entered as a "customer query" by selecting Controlled Substances under CONTEXT Department, not selecting a Service, and then, under Activity, selecting "CS-Customerq". Tracking these communications allows the laboratory to document the need for responsive actions to our client base.

## 4.2 REPORTS

All results should be entered into the approved laboratory computer format for report generation. The report that is issued represents a summary of the analytical findings and should include:

- A. Name of submitting agency
- B. Submitting agency case number
- C. Date of report
- D. Crime Laboratory case number
- E. Name of submitting officer or contributor
- F. Brief description of items analyzed
- G. Weight, volume, or count of each item
- H. Description of any sampling protocols utilized
- I. Results or conclusions
- J. Name of the Forensic Scientist performing analysis
- K. Name of person performing review of findings



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The final report will clearly convey to the officer and/or prosecutor exactly what was analyzed.

Disclaimer information included in all reports under the RESULTS section includes two points:

- “Weights and identifications refer to analyzed items.”
- “Analysis requires the consumption of some of the submitted sample. Reported weights, tablet and capsule counts are pre-consumption quantities.”

Reports should be thoroughly checked by the forensic scientist after they are generated and before sending for review. All reports issued by analysts at the Scientific Crime Detection Laboratory must be subjected to a technical and an administrative review by another qualified forensic scientist prior to issuing the report. See Appendix II **Guidelines for Technical and Administrative Reviews**. Use of this checklist is suggested but not required. Some of the checklist points may not be appropriate in all instances (non-applicable).

A technical review focuses on the analyst's bench notes and the chain-of-custody records. The main purpose of a technical review is to ensure that the conclusions of the examiner are fair and reasonable and based on sound scientific examinations and procedures. The technical reviewer should agree with the conclusions as based on the testing performed, and should be comfortable testifying to the results if the analyst happens to be unavailable for court.

The main purpose of the administrative review is to check for proper transcription of identification numbers, adherence to laboratory policies, proper spelling and grammar, clarity of the report, appropriateness to the agency's request, and distribution of the report to the proper agency or agencies. This last responsibility may be delegated to administrative personnel.

The analyst and the reviewer may consult a third qualified analyst, if necessary, to try to reach an agreement when there is a disagreement on the bench notes or report. If the analysts are unable to come to an agreement, the discipline supervisor will be consulted to make the final decision. If the discipline supervisor is the initial reviewer and there is a disagreement on how to report a result, the laboratory manager will be consulted to make the final decision.

Note: The signature of the reviewer on the final report indicates that the reviewer has performed both an administrative and technical review.

When an error is discovered in a report that has been reviewed and distributed, the discipline supervisor will be notified, the analyst and reviewer consulted, and a corrected

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report issued. If the error is serious enough to question the protocols, policies, or the judgement of the analyst, the Laboratory Quality Manager and Forensic Laboratory Manager will be included in any corrective actions.

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### 4.5 ROUTINE DISCLOSURE

When requests for disclosure (discovery) are received from the prosecutor of a drug case, these will normally be supplied in the form of an 'e-case file' generated in the LIMS and emailed to the appropriate District Attorneys' Office or U.S. Attorneys' Office. This file will include items that are utilized in rendering an expert opinion:

- Final lab reports
- Main case report from LIMS
  - A. General case information
  - B. Agency information
  - C. Offenses
  - D. Individuals
  - E. Chains-of-custody
  - F. Requests for analysis (disciplines needing analysis)
- Requests for Laboratory Services
- Bench notes of analyst
- Spectra from GC/MS or FTIR
  - A. Standards
  - B. Blanks
  - C. Case Samples

This electronic case file is sequentially numbered on the bottom right hand corner. When this disclosure has been provided, documentation of compliance will be the presence of the appropriate e-case file recorded in the LIMS Case Information.

Current Statements of Qualifications (SOQs) for analysts and discipline procedure manuals are available from the laboratory's website.

As per laboratory policy, any requests beyond the scope of routine disclosure require a court order and should be referred to the laboratory's paralegal position and/or the Department of Public Safety's counsel.

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## SECTION 5 REAGENT & STANDARD PROCEDURES

### 5.1 CRITICAL REAGENTS

A reagent log book is used to document reagent preparation and verification. The log book will include:

- A. Reagent name.
- B. Date prepared.
- C. Assigned lot number with initials of person making reagent.
- D. Response to verification standards.
- E. Storage conditions and expiration date (if any).
- F. Initials of person verifying reagent.

Chemical reagents directly affecting case work decisions are verified at the time of preparation or purchase by running a negative (blank) control and a positive (standard) control to safeguard against contamination and ensure proper response. Suggested quality control materials are listed in the reagent log book.

Reagents may be re-tested with known positive and negative controls to assure sensitivity and absence of contamination if any problems arise or at the analyst's discretion. All reagents will be tested every 6 months and results recorded in the Reagent Log Book.

Chemicals used to prepare critical reagents will be purchased from a reputable supplier (approved by laboratory management and listed in a LIMS file named VENDORS), directions for preparation will be adhered to, reagents will be tested when prepared and every six months thereafter with appropriate controls, and documentation will be maintained to record these actions.

Purchase of laboratory chemicals, standards, and supplies is requested on a Laboratory Stock Request Form, given to a supervisor for approval, and then submitted to laboratory management for final approval and budgeting. Examples of order requests for the controlled substances discipline are found on the laboratory network under the Drug\_Share section in a file called ORDERING. The forms need to include descriptive information for each item requested and should be requested from an approved vendor.

The following chemical solutions are considered to be **CRITICAL REAGENTS**, as their performance has a direct effect on casework decisions:

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- **Marquis Reagent**

**Reagent:**

20 mL concentrated sulfuric acid  
16-20 drops formaldehyde (37%)

**Expiration:**

1 month

**QA:**

Check with methamphetamine (orange) or an opiate drug (purple)

**Reference:**

Clarke, E.G.C. *Isolation and Identification of Drugs*, 2<sup>nd</sup> edition; pp. 139-140.

- **para-Dimethylaminobenzaldehyde (p-DAB or Van Urk's)**

**Reagent:**

Dissolve 1.0 gram of para-dimethylaminobenzaldehyde in 100 ml ethyl alcohol and 10.0 ml concentrated HCl.

**Expiration:**

none

**Comment:**

Although this reagent has its primary use as a thin layer chromatography color developing chemical for hallucinogens (ergot alkaloids), it can also be used as a spot test for procaine, psilocyn, and LSD.

yellow  
violet

procaine  
LSD, psilocyn, psilocybin

**QA:**

Check with procaine or psilocyn

**Reference:**

Clarke, E.G.C. *Isolation and Identification of Drugs*, 2<sup>nd</sup> edition; Pharmaceutical Press: London, 1986, p. 132.

- **Scott's**

**Reagent:**

Solution A

2% cobaltous thiocyanate in water and then diluted 1:1  
with 96% USP glycerine

Solution B

Concentrated HCl

Solution C

Dichloromethane

**Expiration:**

none

**Comments:**

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The Scott's test is commercially manufactured into a standard field test for law enforcement officers (Scott Reagent Modified).

"Modified" Scott's reagent is prepared using 10% acetic acid instead of water. This reagent will give a positive reaction for salt or base in the first step.

### References:

1. Scott, L.J., *Microgram*, vol.6, no.11 (November 1973) pp179-181.
2. Prall, J.D., *Microgram*, vol.8, no.9 (September, 1975) pp.130-132.
3. Fasanella, J. and P. Higgins, *Microgram*, vol.19, no.10 (October 1986)

- **Weber Reagent**

#### Reagent:

Solution A

Add approximately 0.01 gram Fast Blue B Salt [o-dianisidine bis (diazotized) zinc double salt] to 10ml water. The solution will have a faint straw color.

Solution B

Concentrated HCl

#### Comments:

The Fast Blue B reagent must be made *fresh* when the mushroom or mushroom extract is ready to be tested. Reagent is suitable for use only on the day it is prepared; at the end of the day the reagent will be appropriately discarded.

#### QA:

Reagent must be tested when made with psilocin or known psilocin containing mushrooms. A red color change after the first step followed by a blue color change after the second step is a positive result.

#### References:

1. Lee, Henry & Harris, Howard, *Physical Evidence in Forensic Science*, 2<sup>nd</sup> Edition, Lawyers & Judges Publishing Co., 2006.
2. San Francisco Police Dept. Criminalistics Lab, *Controlled Substances SOP*, 2005, p.44.
3. Garrett, Clemens, and Gaskill, "The Weber Test: A Color Test for the Presence of Psilocin in Mushrooms", *SWAFS Journal*, Volume 15, Number 1, April, 1993, page 45.

- **Gold Chloride Reagent for Cocaine**

#### Reagent:

5% HAu Cl<sub>4</sub>.3H<sub>2</sub>O

20% Acetic Acid

#### Expiration:

none

#### QA:

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Test with cocaine, hydrochloride or base.

**Reference:**

Butler, W.P. "Methods of Analyses for Alkaloids, Opiates, Marijuana, Barbiturates, and Miscellaneous Drugs"; Internal Revenue Service Publication No. 341: Washington, D.C., 1967.

- **Gold Chloride/Phosphoric Acid Reagent for Methamphetamine**

**Reagent:**

1 gram  $\text{HAuCl}_4$  dissolved in 20 mL of 1:2  $\text{H}_3\text{PO}_4$  in deionized water.

**QA:**

Test with methamphetamine.

**Expiration:**

none

**References:**

1. Aunan, Jayne. Washington State Patrol Laboratory, Spokane, Washington.
2. United Nations Office of Drugs and Crime, "Recommended Methods for the Identification and Analysis of Amphetamine, Methamphetamine, and their Ring-substituted Analogues in Seized Materials", United Nations Publication, p. 44.

- **Silver Nitrate/Cupric Nitrate Microcrystal Test for GHB**

**Reagent:**

100 mg  $\text{AgNO}_3$  and 100 mg of  $\text{Cu}(\text{NO}_3)_2$  dissolved in 10 mL of distilled or deionized water.

**QA:**

Test with GHB primary standard and/or various concentrations of solutions kept in Toxicology freezer.

**Expiration:**

none

**Reference:**

Andera, Kevin, 93<sup>rd</sup> Semi-Annual CAC Seminar, Oakland, CA; May, 1999.

- **Borate Buffer, pH ~9.5**

**Reagent:**

20 grams Boric Acid

5.4 grams NaOH (or 135 mL 1N NaOH or 10.5 mL 50% NaOH)

Dilute to 500 mL with deionized water. Stock bottle under the hood used to refill bench supplies.



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**QA:**

Check pH with 0-13 pH paper. Should be pH 9 to 10.

**Expiration:**

none

- **Chloral Hydrate**

**Reagent:**

50 g Chloral Hydrate

20 mL deionized water

Heat very gently to dissolve, avoid eye and skin contact

**QA:**

Check with known marijuana plant material.

**Expiration:**

none

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### 5.2 STANDARDS

- **Primary Standards, Secondary Standards, Working Standards, and Training Materials**

**Primary Standards** are controlled substances purchased from an approved provider (see VENDORS in LIMS with approved suppliers) or supplied by the DEA or an ISO-accredited crime laboratory. A copy of the current DEA license for purchase of controlled substances is found in a binder kept by the Controlled Substances Supervisor and is also stored electronically on the lab network under Drug\_Share and ORDERING. Records of the purchase of Schedule I and II (federal) drugs are also stored in the binder kept by the Controlled Substances Supervisor. Primary Standards may be a pure (neat) compound or a solution of a neat compound. Any certification documentation accompanying these standards is maintained in a binder in the controlled substances laboratory, but all drug standards must be verified prior to use. Verification is by GC/MS or FTIR analysis. For documentation, see Records, page 53. Primary standards are utilized as reference standards in casework, research and development or methods, training, and quality control of critical reagents.

**Secondary Standards** are comprised of pharmaceutical preparations, plant materials (e.g. marijuana, peyote, commercial synthetic cannabinoid products, salvia, herbs, or mushrooms), some non-controlled drug standards, and selected retained casework samples. They normally do not enter the laboratory with a certification of purity, but are verified at some point by GC/MS or FTIR or are obtained from a reliable manufacturer/distributor or government agency. These standards are utilized for reagent QA (e.g. Weber Test positive control), research where purity is not crucial, training, or confirmation of a non-controlled substance.

**Working Standards** are dilute solutions of drugs prepared for instrumental use or small amounts (less than 500 mg) removed from inventory for QC on critical reagents. The removal of drug from audited inventory is recorded in the binder of drug standards at the time of initial preparation or subsampling, but after that these minute amounts of drug are not tracked for weight.

**Training Materials** are drug substances that are kept in locked storage but not used for any analytical purpose. Training drugs are primarily tablets and capsules, many of them clandestinely manufactured, taken from casework submissions. They are not inventoried and their function is for training and display.

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A "Drug Standards" binder is kept in the controlled substances laboratory with drug standard information including:

- current inventory
- consumed drug standards (or record of destruction/expiration)
- QA verification

The Drug Standards binder is used to document the verification of drug standards. The drug verification should include:

- A. Name of drug, approximate concentration, and diluent.
- B. Date prepared.
- C. Assigned lot number with initials of analyst preparing standard.
- D. Source of standard material with company lot number and/or product identification.
- E. Verification method (GC/MS or FTIR) and the initials of the Forensic Scientist performing verification.
- F. Expiration date (if applicable). Where a purchased chemical or reagent is already marked with an expiration date from the manufacturer, this date shall be effective unless an earlier date is chosen by the laboratory. A later date may also be chosen by the laboratory, but only if it can be demonstrated that there is no loss of performance at the time of usage. A preferable use of 'expired' drug standards is for qualitative training or demonstration exercises.

Drug standards are prepared as needed for instrumental analysis, usually to an approximate concentration of 1mg/mL in methanol. Since these are not used for quantitative analysis, an approximate concentration is sufficient. Each time a new standard is prepared or opened, it receives a lot number, utilizing either the manufacturer's lot number from solution ampules OR a lab-generated lot number, utilizing the convention "MMDDYY analyst initials".

Verification is by GC/MS or FTIR analysis. Primary standards are utilized as reference standards in casework, research and development or methods, training, and quality control of critical reagents.

Standards suspected of being contaminated or decomposed may no longer be used as a standard until re-authenticated.

### • Records

- A. Drug Quality Certificates and any documents of authentication that are received with standards in hard copy form are kept in the **Drug Standards** binder.

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- B.** Verification spectra of new drug standard lot numbers placed into service will be stored in LIMS in the file **CS INST 2011** (or appropriate year) electronically.
- C.** The **Drug Standards** binder stores hard copy records of:
1. Primary Standards inventory
  2. Non-Controlled and Secondary Standards inventory
  3. Consumed drugs
  4. Log of new **working standard** solutions prepared (with QA data) or small amounts (<500 mg) of drug removed for use in quality control of critical reagents.
- Removal of *any* quantity of primary or secondary drug standards from inventory will be recorded in the Drug Standards binder.
- D.** Drug inventory is also recorded electronically at:  
[I:\Discipline Shares\Controlled Substances\Drug Inventories](#)  
on the laboratory's computer network. This is updated yearly after the annual audit.

- **Security**

The Scientific Crime Detection Laboratory is responsible for assuring that its drug standards are secure:

1. All drug standards (controlled or uncontrolled) shall be stored in a locked and secure location.
2. Keys to drug standard locations shall be restricted to the controlled substances discipline supervisor (or designee), the Quality Assurance Manager, and the Laboratory Manager.
3. An audit of all drugs will be made yearly, updates performed, and a memo written to the Lab Manager through the Quality Assurance Manager indicating that this audit was performed. This audit will account for all drugs within the laboratory and will include a yearly gross weight measurement of all controlled substances. The only exceptions to the audit mandate are Working Standards and Training Materials.

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### Appendix I      Abbreviations

**ADA:** Assistant District Attorney

**AgNO<sub>3</sub>:** Silver Nitrate

**APAP:** acetaminophen (for *N*-acetyl-**para**-aminophenol)

**APD:** Anchorage Police Department

**ATR:** Attenuated Total Reflectance

**AuCl<sub>4</sub>:** Gold Chloride

**BATF & E:** Bureau of Alcohol, Tobacco, Firearms, and Explosives

**BB DCM:** Borate Buffer / Dichloromethane

**BD:** 1,4-Butanediol

**BZP:** *N*-Benzylpiperazine

**CBD:** Cannabidiol

**CBN:** Cannabinol

**CH<sub>2</sub>Cl<sub>2</sub>:** Dichloromethane (**DCM** or Methylene Chloride)

**2C-B:** 4-Bromo-2,5-dimethoxyphenethylamine

**2C-E:** 4-Ethyl-2,5-dimethoxyphenethylamine

**2C-I:** 4-Iodo-2,5-dimethoxyphenethylamine

**2C-T-7:** 2,5-Dimethoxy-4-(n)-propylthiophenethylamine

**Cu(NO<sub>3</sub>)<sub>2</sub>:** Cupric Nitrate

**DA:** District Attorney

**DAO:** District Attorney's Office

**DCM:** Methylene Chloride (**CH<sub>2</sub>Cl<sub>2</sub>** or Dichloromethane)

**DIB:** Drug Identification Bible

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**DiP-5-MeOT:** N,N Diisopropyl-5-methoxytryptamine

**DiPT:** N,N-Diisopropyltryptamine

**DOC:** 2,5-Dimethoxy-4-chloroamphetamine ( or 4-Chloro-2,5dimethoxyamphetamine)

**DOB:** 2,5-Dimethoxy-4-bromoamphetamine (or 4-Bromo-2,5-dimethoxyamphetamine)

**DOET:** 2,5-Dimethoxy-4-ethylamphetamine

**DOI:** 2,5-Dimethoxy-4-iodoamphetamine

**DOJ:** Department of Justice

**DPT:** N,N-Dipropyltryptamine

**d. WATER:** distilled or deionized water

**e-Case File:** Electronic Case File

**EtOH:** Ethanol

**FTIR:** Fourier Transform Infrared Spectroscopy

**FPD:** Fairbanks Police Department

**FBKS:** Fairbanks

**g:** gram

**GC/MS (or GC-MS):** Gas Chromatography-Mass Spectrometry

**GHB:** Hydroxybutyric Acid

**GBL:** Gamma Butyrolactone

**HAuCl<sub>4</sub>:** Gold chloride

**HCl:** Hydrochloric acid

**HCO<sub>3</sub>:** Bicarbonate

**HI:** Hydriodic Acid

**H<sub>2</sub>O:** Water

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**4-HO-DIPT:** 4-Hydroxy-N,N-diisopropyltryptamine

**HP:** Hewlett Packard

**H<sub>2</sub>SO<sub>4</sub>:** Sulfuric Acid

**H<sub>3</sub>PO<sub>4</sub>:** Phosphoric Acid

**ICFI:** Insufficient Concentration for Identification

**IPA** – Isopropyl Alcohol (or Isopropanol)

**IR:** Infrared Spectroscopy

**ISFI:** Insufficient Sample for Identification

**ID:** Identification

**JWH-018:** 1-naphthalenyl-(1-pentyl-3-indolyl)methanone

**JWH-073:** (1-butyl-3-indolyl)-(1-naphthalenyl)methanone

**JWH-250:** 2-(2-methoxyphenyl)-1-(1-pentyl-3-indolyl)ethanone

**KBR:** Potassium Bromide

**kg:** kilogram

**lb(s):** Pound(s)

**Less than:** <

**LIMS:** Laboratory Information Management System

**LSD:** Lysergic acid diethylamide

**5-MeO-DIPT :** 5-Methoxy-N,N-diisopropyltryptamine

**5-MeO-DMT:** 5-Methoxy-N,N-dimethyltryptamine

**5-MeO-MIPT:** 5-Methoxy-N-methyl-N-isopropyltryptamine

**5-MeOT:** 5-Methoxytryptamine

**6-MeOT:** 6-Methoxytryptamine

**MeOH:** Methanol



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**MDA:** Methylenedioxyamphetamine

**MDMA:** Methylenedioxymethamphetamine

**MDOH:** N-Hydroxy-3,4-methylenedioxyamphetamine

**mg:** Milligram

**mL:** Milliliter

**MMDA:** 5-Methoxy-3,4-methylenedioxyamphetamine

**MMDDYY or MMDDYYYY:** 2 digit month/2 digit day/ 2 or 4 digit year

**MSD:** Mass Selective Detector

**MSDS:** Material Safety Data Sheet

**MSM:** Methylsulfonylmethane or Dimethyl Sulfone

**MSTFA:** N-methy-N-(trimethylsilyl) trifluoroacetamide (or N-trimethylsilyltrifluoroacetamide)

**5-MT:** 5-Methyltryptamine

**7-MT:** 7-Methyltryptamine

**µl or µL:** Microliter

**N/A:** Not Applicable

**NA:** Not Analyzed

**NaOH:** Sodium Hydroxide

**Na<sub>2</sub>SO<sub>4</sub>:** Sodium Sulfate

**NCSD:** No Controlled Substances Detected

**NH<sub>4</sub>OH:** Ammonium Hydroxide

**NFLIS:** National Forensic Laboratory Information System

**NIST:** National Institute of Standards and Technology

**NSB PD:** North Slope Borough Police Department

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**pck:** Package

**PD:** Police Department

**PDR:** Physicians' Desk Reference

**p-DAB:** P-Dimethyl aminobenzaldehyde

**Pet Ether:** Petroleum Ether

**PFTBA:** Perfluorotributylamine

**PMA:** 4-Methoxyamphetamine

**P-2-P:** Phenyl-2-propanone

**QA:** *Quality Assurance*

**QC:** *Quality Control*

**QIFA:** Quantity Insufficient for Analysis

**QNS:** Quantity Not Sufficient

**SOQ:** Statement of Qualifications

**STP:** 2,5-Dimethoxy-4-methylamphetamine

**TFMPP:** Trifluoromethylphenylpiperazine

**THC:** Tetrahydrocannabinol

**TLC:** Thin Layer Chromatography

**Triage:** The practice or principle of allocating limited resources; prioritization

**UAA:** University of Alaska Anchorage

**UAF:** University of Alaska Fairbanks

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### Appendix II

### Weight Considerations in the Alaska Statutes

- MICS I      11.71.010
  - 1) delivers **any amount** of IA
  - 2) delivers **any amount** IIA or IIIA to person <19 years old
  
- MICS II      11.71.020
  - 1) manufactures or delivers **any amount** IA
  - 2) manufactures any **amount** of methamphetamine
  - 3) possesses more than **6 grams** of ephedrine or pseudoephedrine with intent to manufacture
  
- MICS III      11.71.030  
  IIIA
  - 1) manufactures or delivers **any amount** of IIA or
  - 2) delivers **any amount** of IVA, VA, or VIA to a person less than 19 years old
  
- MICS IV      11.71.040  
  VA
  - 1) manufactures or delivers **any amount** of IVA or
  - 2) manufactures or delivers a weight of **1 ounce** or greater VIA
  - 3) possesses **4 ounces** or greater of VIA
  
- MICS V      11.71.050  
  VIA
  - 1) manufactures or distributes **less than 1 ounce**
  - 2) possesses **greater than 3 grams** IIIA or IVA
  - 3) possesses **greater than 1 ounce** VIA
  
- MICS VI      11.71.060
  - possesses **less than 1 ounce** VIA

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### Appendix III Guidelines for Technical and Administrative Reviews

<b>TECHNICAL REVIEW</b>	<b>Analyst:</b>	<b>Date:</b>
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Yes N/A

- ☐ ☐ The correct laboratory number appears on each page of the case file.
- ☐ ☐ The analyst's initials or name appear on all pages of the case file.
- ☐ ☐ Sub-itemizations are clearly labeled and consistent throughout LIMS, bench notes, and report.
- ☐ ☐ For each controlled substance identified, a standard was analyzed and is included in the notes.
- ☐ ☐ For each GC/MS or FTIR evidence spectra, a blank is included in the notes.
- ☐ ☐ Evidence retention times and spectra agree with standard retention times and spectra.
- ☐ ☐ 'Physical identifications only' have adequate documentation.
- ☐ ☐ All reference or internet searches are documented in the case file.
- ☐ ☐ The worksheet indicates the dates that analysis was started and completed.
- ☐ ☐ Each item's packaging is documented.
- ☐ ☐ The officer's description of items agrees with the analyst's description.
- ☐ ☐ The weights or counts on the worksheet and the report are in agreement and the weights are truncated in the report.
- ☐ ☐ The type of analysis and results are documented for each item in the bench notes.
- ☐ ☐ All chemical testing and all physical identification searches are documented in the notes.
- ☐ ☐ Two samplings are used for all conclusive identifications.
- ☐ ☐ An appropriate extraction/clean-up procedure was employed.
- ☐ ☐ The analysts' conclusions in the report are supported by documentation in the notes.
- ☐ ☐ The item descriptions agree between LIMS, bench notes, spectra, and report.
- ☐ ☐ The electronic chain of custody in LIMS agrees with the worksheet and report.

<b>ADMINISTRATIVE REVIEW</b>	<b>Analyst:</b>	<b>Date:</b>
------------------------------	-----------------	--------------

Yes N/A

- ☐ ☐ The requesting agency, agency file number, and lab number agree with the lab submittal.
- ☐ ☐ The correct name of the submitting officer and/or whom to reply to appears on the report.
- ☐ ☒ The lab item number and description of the item are correct.
- ☐ ☒ Items examined but not analyzed are included in the report Description & Results.
- ☐ ☐ Grammar, spelling, and punctuation are correct.
- ☐ ☐ The report is signed by the analyst.
- ☐ ☐ Any latent print request is changed from "Latents Pending" to "Latents Processing" and re-dated.
- ☐ ☐ Report is referred appropriately for distribution to agency.

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### Appendix IV Hypergeometric Sampling Plan

# of UNITS	SCREEN & CONFIRM
1-10	All
11-12	10
13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-939	28
940+	29

#### Hypergeometric Sampling

- Based on statistical probability, there is a 95% likelihood that at least 90% of the units contain the drug
- Visual similarity adds another level of confidence. If a sampling plan is applied, the expert's opinion is that all units are the same substance.
- After confirming 29 units, there is no significant increase in confidence level.
- 29 units = ~6 hours of instrument time
- If any results are different than the rest, The analyst must re-evaluate the population.

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### Appendix V      Uncertainty of Measurement

(Policies to be determined)

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### Appendix VI      SWGDRUG Recommendations

See [I:\Discipline Shares\Controlled Substances\SWGDRUG Guidelines](#)

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### Appendix VII Revision History

Section(s) Revised	Date	Issuing Authority
<b>Table of Contents</b> , p. 1, changed "Calling Criteria" to "GC/MS Interpretation of Data and Criteria for Identification by GC/MS".	1-23-2012	Jane Booth, Supervisor
<b>Section 3.1, GC/MS, Interpretation...</b> , p. 34, added " <b>GC</b> " resolution, sensitivity, and peak shape "on the total ion current chromatogram (TIC) and acceptable <b>mass spectra</b> for the cocaine peak". <u>Removed</u> interpretation guidelines for other 3 compounds and <u>removed</u> last sentence "Note that only one of the four components of the QC Mix is recorded in LIMS, however, the analyst is to check <i>all</i> components and rotate the drug recorded monthly."	1-23-2012	Jane Booth, Supervisor
Changed <b>Laboratory Computer Network</b> address to I:\Discipline Shares\Controlled Substances\....., pp. 29, 36, 39, 40, 54, & 64.	1-23-2012	Jane Booth, Supervisor
<b>Appendix III Guidelines for TR and AM</b> , p. 61, added to Administrative Review. "Items examined but not analyzed are included in the report Description and Results".	1-23-2012	Jane Booth, Supervisor